REVISTAMATERIA V.23 N.03

ISSN 1517-7076 artigo e-12187, 2018

Physico-chemical characterization of centrifuged sludge from the Tamanduá water treatment plant (Foz do Iguaçu, PR)

Kleber Gomes Ramirez[1], Edna Possan[2],
Paulo Rodrigo Stival Bittencourt [4], Charles Carneiro[3], Mauricio Colombo[4]

[1] Water and Sanitation Company of Paraná State (SANEPAR), CEP: 80215-900, Curitiba, PR, Brazil.
[2] Graduate Program in Civil Engineering, Federal University of Latin American Integration Federal (PGCEI/UNILA), CEP: 85867-000, Foz do Iguaçu, Paraná, Brasil.
[3] Institute of Business Administration and Economics (ISAE/FGV) and Water and Sanitation Company of Paraná State (SANEPAR), CEP: 80215-900, Curitiba, Paraná, Brazil.
[4] Federal University of Technology, UTFPR , CEP 85884-000, Medianeira, Paraná, Brazil.
e-mail: kleber_r@hotmail.com, epossan@gmail.com, paulob@utfpr.edu.br, charlesc@sanepar.com.br, colomboecia@hotmail.com

ABSTRACT

The water treatment process generates a residue called water treatment plant (WTP) sludge, which needs to be correctly characterized to ensure appropriate disposal or reuse. This study aimed to characterize the centrifuged sludge produced at the Tamanduá WTP, Iguaçu Falls City, Brazil, and considered opportunities for its reuse in the production of concrete for the civil construction industry. Wet sludge (sludge in its natural form) analysis included the determination of total solids, moisture content, density, pH, and inorganic parameters (As, Al, Ba, Cd, Pb, Cr, F, Hg, Ag, and Se) through thermogravimetric analysis, X-ray diffraction, and chemical analysis by X-ray fluorescence and loss ignition. For calcined WTP sludge, chemical and mineralogical composition and laser granulometry were evaluated. The results indicated that calcined sludge had the potential to be used in the production of cement materials; conversely wet sludge did not reach the appropriate safety standards due to the high quantity of organic matter.

Keywords: Water treatment plant, water sludge, coagulant, solid waste.

1. INTRODUCTION

The growing demand for potable water and the increasing pollution of water sources have a direct impact on the production of water treatment plant (WTP) sludge in the water treatment process. In Brazil, this subject requires further discussion and research [1]. Identifying new treatment methods and handling processes, as well as appropriate final destinations for this waste, is a challenge for engineers and researchers all over the world [2, 3]. It is necessary to find alternative uses for the waste generated by the production processes, which are technical and economically feasible, and minimize the environmental impact [4].

Most Brazilian WTPs use a conventional process for water treatment due to high turbidity and color, and the presence of colloidal matter [5]. Generally, the conventional water treatment process includes coagulation, flocculation, decantation, filtration, disinfection, and fluoridation. This process consists of the addition of iron or aluminum salt, which destabilizes the colloidal particles in solution and in suspension in the raw water. The particles form flakes, which are sedimented in decanters and then filtered for the final clarification, generating the WTP sludge [6]. Depending on the treatment process, the WTP sludge may be either densified (mechanical process), centrifuged (mechanical process), or dehydrated (physical process). Each of these processes affect the moisture content of the sludge produced.

Among all Brazilian municipalities (5,570 cities), 37.7% (2,098 cities) generate WTP sludge, with 67.4% (1,415 cities) of these disposing the waste to rivers, generally without any type of treatment [5]. In Brazil, there is an average production of 762,500 tons/day of sludge from conventional WTP and 2,000 tons/day of sludge from WTP without any treatment [7]. Data from the Water and Sanitation Company of Paraná State (SANEPAR) [8] indicates that the total treated water volume in its 162 WTPs generates 17,000 tons of dry matter a year from centrifuged sludge.

According to Brazilian Standards, WTP sludge is classified as solid and semi-solid waste and must be treated and disposed of as required by the regulatory authorities in compliance with the National Solid Waste Policy [10]. Hence, sanitation companies in the sector have been seeking alternative and environmentally-friendly solutions for the

Autor Responsável: Edna Possan Data de envio: 15/07/2017 Data de aceite: 06/03/2018

10.1590/S1517-707620180003.0521

disposal of the waste produced in the water treatment process.

In addition to the environmental impact where the WTP waste is disposed, the sludge can also pose a risk to human health due to the presence of pathogenic agents and heavy metals [11]; thus appropriate disposal or reuse of this waste is important. According to Tsutiva and Hirata [12], the main challenge is the need for further research into alternatives for the disposal of WTP sludge that are economically and technically feasible, and advantageous to the environment.

In recent years, many researchers have studied the use of WTP sludge [28] in degraded areas [13, 14], coagulant regeneration [15], and reuse in the construction industry as a replacement for, or addition to, the traditional raw materials in the production of cement [16, 17, 18], concrete [3, 11, 19, 20, 21, 22], ceramic [23, 24, 25, 26, 27, 44], soil-cement [29], and mortars [30, 31].

However, existing treatment and disposal methods have rarely been adopted in Brazilian WTPs, which is mainly due to high costs [32] as well as due to inconsistencies in research [3], which highlight the environmental and economic importance of developing alternatives. According to Wang et al. [33], there are technologies that can reduce the amount of sludge generated in the water treatment process.

In addition, WTP sludge composition often varies and is directly related to the characteristics of the water source used [1]. The sludge can contain distinct substances in various concentrations due to the inherent characteristics of the surrounding watershed (geological substrate, soil type, forest type, and topography), soil use, climatic factors (primarily rainfall intensity), and the type of coagulant used in the water treatment process, which varies according to the seasonal characteristics of the sludge [1, 4]. The qualitative and quantitative sludge characteristics can also vary according to the management of the treatment process, system operation methods, frequency of decanter and filter cleaning, and chemical dosages [23]. In addition, WTP sludge that is densified or centrifuged has a high moisture content and it is therefore necessary to use other technologies for its drying and subsequent reuse [3].

Since the WTP sludge does not exhibit a regular composition or behavior, the dehydration process becomes more difficult. Moreover, given a lack of technical research into sludge treatment and disposal, it is difficult to develop custom treatments that are appropriate and economically viable [1]. The first step in developing appropriate treatment and disposal methods is the assessment and characterization of the water sludge, which is the aim of the present study.

2. MATERIAL AND METHODS

2.1 Research site and WTP sludge production

This study was carried out using sludge from the Tamanduá WTP (Figure 1), located in Iguaçu Falls City, Paraná state (25º34' south, 54º31' west). The sludge was collected between January and March 2014.

The Tamanduá River, which gives its name to the WTP (Figure 1), has a current operational flow rate of 900 m³/hour; this limit is set by a concession granted by the Water Institute [8]. The Tamanduá River is the main watercourse in the watershed, which has an area of approximately 145 km². The Tamanduá WTP produces approximately 21,600 m³ of potable water per day. The annual intake volume, volume of potable water produced, and the waste mass produced (WTP centrifuged sludge) for the previous 10 years are shown in Table 1 [8].

Figure 1: Tamanduá WTP - Iguaçu Falls. Source: Google Earth [34] e Kleber Ramirez (arquivo pessoal).

Table 1: WTP sludge production, intake volume, and potable production volume at Tamanduá WTP (Iguaçu Falls, PR) [8].

YEAR	WTP SLUDGE	WATER VOLUME (m³)

	(kg)	INTAKE	PRODUCED
2007	45,070	6,924,343	6,816,859
2008	265,740	6,694,461	6,646,969
2009	39,840	6,505,567	6,462,503
2010	37,250	5,845,535	5,813,202
2011	116,890	6,119,668	6,056,022
2012	85,230	6,199,162	6,154,314
2013	151,660	7,119,652	7,025,625
2014	101,580	7,425,034	7,260,592
2015	31,421	7,470,432	6,943,827
2016	57,280	7,992,634	7,353,439

The water treatment system at Tamanduá WTP comprises 2 modules, 6 decanters, 12 filters, and 2 hydraulic flocculators, and has a treatment capacity of 250 L/second. The water treatment starts with the intake of raw water from the Tamanduá River, followed by a pre-chlorination process performed according to the Daily Treatment Schedule (DTS), pH correction, and alkalinity control using hydrated calcium oxide (CaO) as necessary. Once complete, aluminum polychloride ($Al_n(OH)_mCl_{3n-m}$) coagulant is added in the *parshal* flume for rapid agitation, followed by flocculation through slow agitation. Water then flows to the decanter, where the solid particles decant and the clarified water passes to the filtering process. The filtered water then flows to the contact tanks, where the disinfection process is performed using chlorine gas (Cl_2) and the addition of fluorine (Na_2SiF_6). The final treated water is then distributed to the local population (see Figure 2).

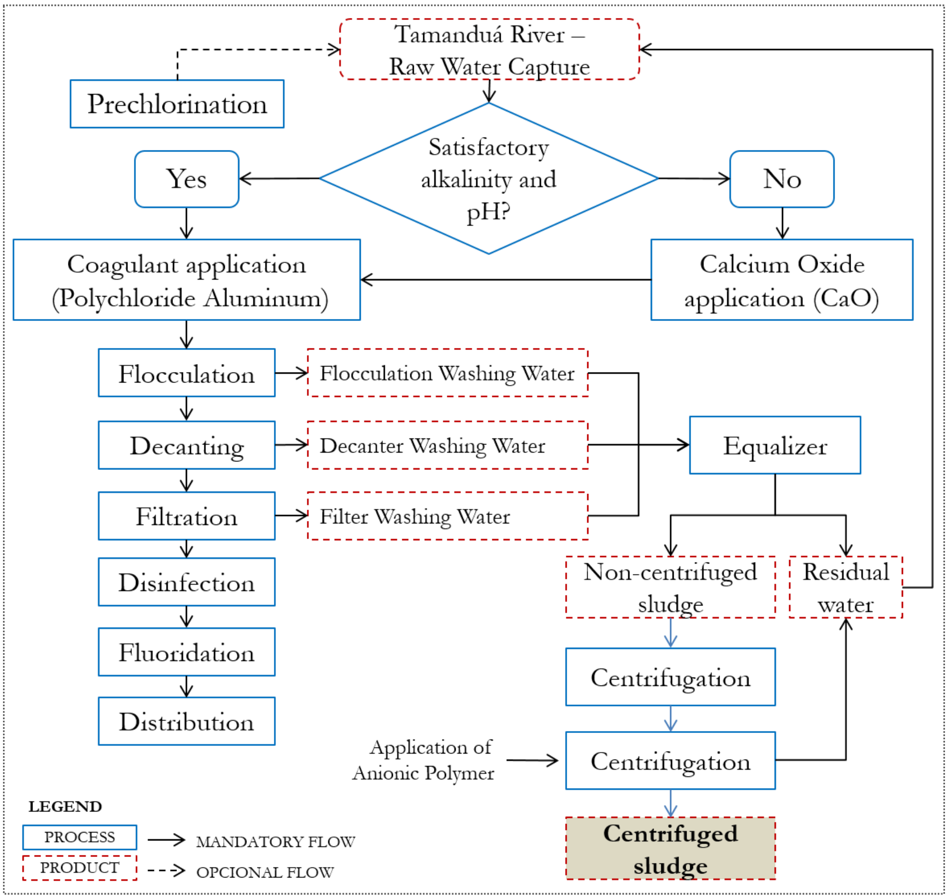


Figure 2: Tamanduá WTP flowchart [35].

Figure 3: WTP sludge in the thickening tank.

Figure 4: WTP sludge after centrifugation.

The wash water from the flocculation tank, decanter, and filters is driven to the equalization tank, where the decanted sludge is pumped to the thickening tank and the supernatant material is recirculated to the beginning of the process. The decanted solids retained at the bottom of the decanters follow to the sludge thickening tank (Figure 3) and subsequently to the centrifuged decanter, in which an anionic polymer is added to form the sludge cake. The liquid portion is returned to the treatment process via recirculation and the drier portion (sludge) is taken by road to landfill. Figure 4 shows the WTP sludge in the thickening tanks, where the samples used in this study were collected.

2.2 Sludge collection

Sludge samples were gathered over 3 periods in 2014, with 4 samples collected in each period: Period 1: January–March; Period 2: June–July; and Period 3: August–October. The periods chosen took account of preceding weather conditions (precipitation, wind, and temperature) and the possible influence of the weather on the samples was investigated. The samples were stored in acrylic containers with a lid (Figure 5) until homogenization was performed (Figure 6) so as to preserve the sludge characteristics.

Figure 5: Sludge sample storage.

Figure 6: Sludge homogenization

2.3 Sludge Characterization

The characterization tests performed on the Tamanduá WTP sludge are shown in Table 2

Table 2: Summary of sludge characterization tests.

STEPS	ANALYSIS	METHOD/EQUIPMENT	REFERENCE
Preliminary tests	Moisture content	Stove	Embrapa [36]
	Total solids	Gravimetric method	NBR 10664 [38]
	Density	Volumetric ring	Embrapa [36]
	Hydrogen potential - pH	Potentiometrically method	NBR 10004 [9] e 10005 [37]
Material characterization	Inorganic parameters	-	APHA [36]
	Chemical composition	XRF	-
	Mineralogical composition	XRD	-
	Thermogravimetric	TGA	-
	Average diameter	Laser diffraction	-

2.3.1 Determination of moisture content, density, total solids, and pH

Determination of sludge moisture content was performed by oven drying (110°C), while density and total solids were calculated using volumetric ring and gravimetric methods, respectively. The sludge pH was determined potentiometrically using leached extract analysis following NBR 10005 [37] and NBR 10004 [9] standards.

2.3.2 Chemical analysis of inorganic parameters

Analysis of inorganic parameters was performed according to the Standard Methods for Examination of Waste and Wastewater [36].

2.3.3 Thermogravimetric analysis

Thermogravimetric analysis (TGA) and differential thermogravimetric (DTG) analysis were performed in temperatures ranging from 50°C to 900°C, using simultaneous thermal analysis (STA) equipment (STA 6000, PerkinElmer) with an opened platinum crucible and oxygen atmosphere with a flow rate of 100 mL/min^{-1} and an oven heating rate of 10°C/min^{-1}.

2.3.4 X-ray Fluorescence (XRF)

Analysis of sludge chemical composition was carried out using an X-ray fluorescence spectrometer (Axios Max, PANalytical) with SuperQ51 interpretation software. The fundamental parameters (FP) method was used for semi-quantitative determination. The preparation procedure involved pressed pellets (sample and organic wax), a loss ignition test at 1,000ºC, and a chemical scan.

2.3.5 X-ray diffraction (XRD)

Mineralogical composition was obtained by XRD using a diffractometer (EMPYREAN, PANalytical), operated at 40 kV and 40 mA, and utilizing Cu-Kα (λ = 1,54060 Å) radiation. The angular speed was 10 min^{-1} and a scanning interval of 2θ. The data from the sample interplanar spacing (d-spacing) was compared with available standards from the International Centre for Diffraction Data/Joint Committee on Power Diffraction Standards (ICDD/JCPDS).

2.3.6 Granulometry distribution

Granulometry analysis by laser diffraction was performed using a Cilas DB1 analyzer, reusing alcohol as the particle dispersing agent.

3. RESULTS AND DISCUSSIONS

3.1 Moisture content, total solids, density, pH, and inorganic parameters

Table 3 presents the values for moisture content, density, and total solids for the 12 WTP sludge samples collected in the 3 sampling periods. Analysis of variance (ANOVA) showed that there was no significant difference between the collected samples (Period 1, 2, and 3) for each of the 3 analyses under investigation. This indicated that the WTP sludge presented similar characteristics throughout the study period. Therefore, the sludge was considered as one single sample, produced through the homogenization of all the collected samples.

The average moisture content was 76% (approximately 24% total solids), which was a similar value to that found by Tartari [23] in a study of the same WTP in 2011 (average moisture content of 74% and total solid content of 26%). Tafarel et al. [22] identified a moisture content of 86% in thickened sludge. According to Richter [39], this percentage is considered satisfactory for mechanical dehydration by centrifugation, with total solids varying between 16% and 35%.

The moisture content has direct implications for sludge disposal or reuse. In relation to the application of WTP sludge in the production of concrete, Ramirez [35] observes that the water quantity must be strictly controlled due to its negative influence on the concrete mechanical proprieties, thus limiting its utilization in concrete production. The author also emphasizes that there are many techniques for sludge dewatering (thickening, centrifugation, etc.) that could be used at WTPs, reducing the moisture content and sludge volume to be transported.

The average density (ρ) of the wet sludge was 1.17 g/cm³ (SD± 0.013), a value much closer to that of water due to its high moisture content (76%). This result corresponds with literature [39], which establishes a density of 1.061 to 1.189 g/cm³ for centrifuged sludge with 25% total solids. In contrast, for the thickened WTP sludge, Tafarel et al. [22] found a slightly higher density (1.25 g/cm³), which was possibly due to a lower moisture content from the sample.

As for the total solids, the average value found (23.63%) was within the limits recorded in literature [22, 32, 39]. According to Richter [39], for sludge dehydration by centrifuge, the total solids varies between 16% and 35%. In relation to pH, the studied sludge had alkaline characteristics, with a hydrogen potential of 7.9. Yague at al. [21] found a pH of 7.08, while Tafarel et al. [22] found values of 6.8 and 6.7 for discharged and thickened sludge, respectively, and therefore less alkaline than the sludge used in the present study. Table 4 shows data from the leachate analyses. The values recorded were within the limits set out in NBR 10.004 [9].

Table 3: Moisture content, density, and total solids.

COLLECTION	SAMPLE	MOISTURE CONTENT (%)	DENSITY (g/cm³)	TOTAL SOLIDS (%)
1	1	74.94	1.21	25.05
	2	66.76	1.22	33.24
	3	80.31	1.13	19.69
	4	80.32	1.18	19.68
	Average	**75.582**	**1.185**	**24.415**
	SD	**6.404**	**0.0404**	**6.404**
2	1	74.44	1.18	25.55
	2	78.75	1.19	21.25
	3	78.06	1.11	21.94
	4	79.52	1.23	20.48
	Average	**77.692**	**1.1775**	**22.305**
	SD	**2.249**	**0.0499**	**2.244**
3	1	78.5	1.09	21.49
	2	80.02	1.17	19.98
	3	71.49	1.15	28.5
	4	73.29	1.2	26.71
	Average	**75.825**	**1.1525**	**24.17**
	SD	**4.081**	**0.0465**	**4.08**
Sample average		**76,366**	**1.172**	**23.63**
Standard Deviation		**4,071**	**0.042**	**4.069**

Table 4: Sludge inorganic parameter.

ANALYTE	DETECTION LIMIT (mg/L)	LEACHATE MAXIMUM LIMIT[1] (mg/L)	RESUTS (mg/L)
As	0.0001	1.0	<0.0001
Al	0.01	NA[2]	8.98
Ba	0.005	70.0	<0.005
(Cd)	0.006	0.5	0.04
(Pb)	0.01	1.0	0.63
(Cr)	0.05	5.0	0.02
(F^-)	0.01	150.0	0.37
(Hg)	0.0001	0.1	<0.0001
(Ag)	0.001	5.0	0.005
(Se)	0.001	1.0	<0.001

Note: [1] Leachate maximum limit according to ABNT NBR 10.004 [9]. [2] N/A = Not applicable.

It is not uncommon to observe high amounts of heavy metals in sludge. They come from products used in the treatment process, such as aluminum polychloride, aluminum sulphate, ferrous sulphate, and sodium aliminate, and they have a direct effect on the chemical composition of the WTP sludge. Martínez-García et al. [25] studying the WTP sludge from Jaen (southern Spain) observed high values of iron (Fe) (2.11%) and aluminium (Al) (2.87%) due to the addition of flocculating agents, as well as the presence of calcium (Ca), magnesium (Mg) and sodium (Na), which was likely to have been supplied by sediments from the urban sewage system.

Tsutiya [12] suggests that the sludge characterization should be linked to the preferred final disposal destination and not only set by the characterization parameters established by NBR 10.004 [9]. The author also emphasizes that the parameters analyzed allow only a general evaluation of the sludge quality, indicating possible uses; however, some parameters could be omitted and/or added.

3.2 Thermogravimetric analysis

TGA of the WTP sludge is shown in Figure 7, which reveals at least 3 distinct steps (S1, S2, and S3) of mass loss with increasing temperature. This characteristic has also been observed by Pinheiro [40]. Figure 8 shows the mass loss interval of steps S2 and S3. The DTG curve shows two peaks of mass loss, T' and T'', whose temperature range was 180ºC to 300ºC and 370ºC to 500ºC, respectively.

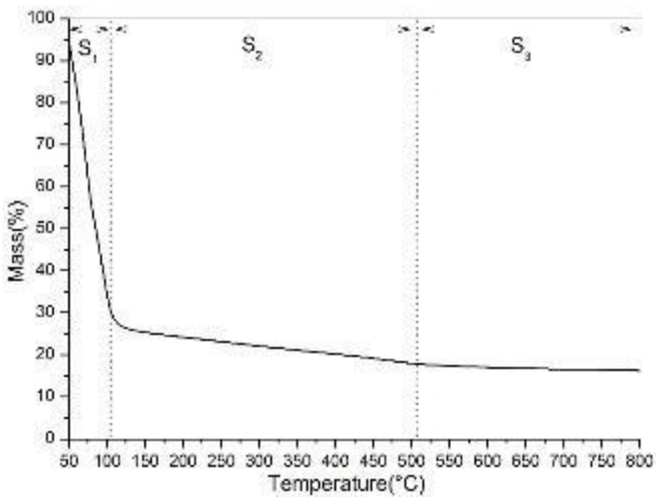


Figure 7: Sludge thermogram (TGA).

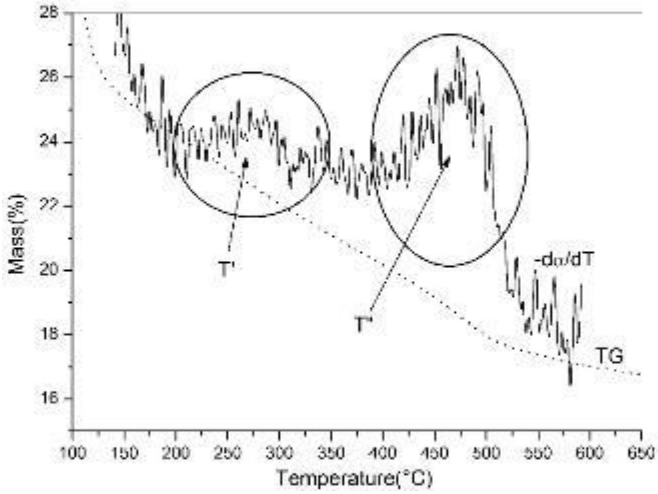


Figure 8: Thermogram (TGA) and differential thermogravimetric (DTG) analyses of the sludge sample.

The second step (S2), whose temperature range was 110ºC to 500ºC, involved the release of volatile compounds and degradation of organic compounds [11, 40]. The mass content released from the sample at this step was approximately 9.5%. Gastaldini et al. [11] related the mass loss between 200ºC and 400ºC with an exothermal peak corresponding to the decomposition of organic matter. The last step (S3) of mass loss (temperature between 500ºC and 800ºC) was characterized mainly by carbon dioxide (CO_2) release due to the degradation of the carbonates formed at S2 and the release of other non-metallic oxides (to a smaller extent). At the end of the test, the residual mass content, which was composed mainly of inorganics, was around 16% of the initial mass.

In the first step (S1), with temperatures from 0ºC to 110ºC, it was observed that the mass loss initiated at the beginning of the test was characterized by the release of the sample moisture (free moisture) [32]. In this step, the residual mass content was 27%, indicating that 73% of the sample mass was water. This initial mass loss was expected considering that the sludge did not undergo prior heat treatment and the moisture content analysis, performed by oven method, indicated a moisture content of approximately 76%.

3.3 Chemical composition by XRF

XRF analysis (Table 5) showed that the chemical composition of the WTP sludge included higher concentrations of aluminum (Al_2O_3), silicon (SiO_2), and iron (Fe_2O_3) oxides. The sum of SiO_2, Al_2O_3, and Fe_2O_3 in the matrix corresponded to approximately 69.9% of the total chemical components for the wet sludge and 92.6% for the calcined sludge. These values are similar to those found by Tartari [23] for the same oxides for wet sludge (74%) (Table 6).

Table 5: Chemical composition of wet and calcined sludge.

COMPOSITION (%)															SAMPLE TYPE
SiO_2	Al_2O_3	Fe_2O_3	TiO_2	SO_3	P_2O_5	CaO	MgO	MnO	K_2O	Cl	V_2O_5	Na_2O	ZrO_2	LOI	
27.3	24.2	17.5	2.35	0.4	0.3	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1	27.1	Wet sludge
38.6	33.6	20.4	3.4	0.1	0.3	0.3	0.3	0.2	0.2	-	0.1	0.1	0.1	2.47	Calcined*

* Calcined at 700ºC. LOI = loss on ignition.

Table 6: Chemical composition of wet sludge from different water treatment plants.

AUTHORS	COUNTRY	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	LOI
Yague et al. [21]	Spain	29.70	12.90	10.10	22.7	2.73	1.83	0.23	-
Lin et al. [41]	Taiwan	53.60	20.90	6.60	0.30	1.90	2.90	0.97	-
Vieira et al [27]	Brazil	14.49	20.19	6.23	0.13	-	0.14	-	57.73
Chen et al. [16]	China	52.10	19.90	6.29	1.68	1.38	2.90	0.97	-
Rodríguez et al.[31]	Spain	29.60	17.57	5.18	11.8	2.15	2.85	6.09	22.00
Tartari et al. [23]	Brazil	24.10	31.60	18.60	-	-	0.30	-	20.40

Martínez-Garcia et al. [25]	Spain	46.37	30.33	8.55	11.15	2.19	3.25	0.36	-
Wolff et al. [42]	Brazil	37.50	30.10	12.30	0.20	0.40	0.90	0.20	17.1
Gastaldini et al. [11]	Brazil	66.02	17.7	8.76	0.57	0.96	1.16	0.32	3.37*
Pinheiro et al. [40]	Brazil	26.84	26.33	24.00	0.14	0.32	0.34	0.03	19.32
Andrade et al. [4]	Brazil	15.60	31.10	6.60	0.30	0.10	0.20	0.30	44.49

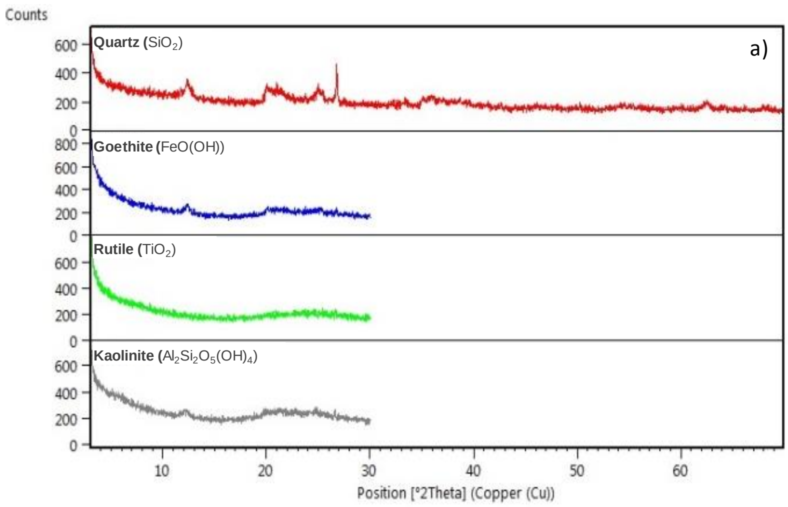
LOI: Loss on Ignition. *Calcined at 600ºC.

The high concentration of SiO_2 could be attributed to the composition of the material sedimented in the water treatment process and was mainly due to the presence of kaolinite [40, 44]. The presence of Al_2O_3 could be related to the coagulant used in the water treatment process (polyaluminum chloride (PAC)), which directly reflected the chemical composition of the sludge and has been observed in other literature [27, 40]. Studies from Spain [21, 25, 31] show calcium oxide (CaO) values from 11.2% to 22.7% in sludge (Table 6). These results indicate that the chemical characteristics of different WTP sludge can be related to local geological characteristics (e.g. watershed adduction) and to the coagulant used in the water treatment process adopted by the WTP.

The presence of alkaline oxides (K_2O and NaO_2), alkaline earth metals (MgO and CaO), titanium oxide (TiO_2), and phosphorus pentoxide (P_2O_5) were due to the use of coagulants in the water treatment process and the water composition [27], which contained suspended materials as sand and clay particles. With regards to the high value of Fe_2O_3, this could be related to the presence of goethite (iron hydroxide (FeO(OH)) and hematite (iron oxide (Fe_2O_3)) in the sludge (see Figure 9). Regarding the loss on ignition, a value of 27.05% was achieved for the wet sludge. This high value was possibly due to the presence of zeolite interstitial waters, hydroxyls of clay minerals, and existing hydroxides. It could also be partly due to volatile organic matter components found in the wet sludge. Table 6 shows that the loss on ignition from different sludges ranged from 17% to 57.7%, which corresponds with the results from other studies of wet sludge [23, 31, 40, 42]. The loss on ignition for the calcined sludge was 2.47%, a value similar to that found by Gastaldini et al. [11] for a sludge subjected to heat treatment at 600ºC (Table 6). This value is within the required range established by Brazilian regulation [43], which recommends that pozzolanic materials must meet a maximum loss on ignition of 6%.

3.5 XRD and granulometry

The diffractogram for wet sludge shown in Figure 9a identifies the peak characteristics of the crystalline phases of quartz minerals (SiO_2), goethite [FeO(OH)], and rutile (TiO_2), and clay minerals from the kaolinite [$Al_2Si_2O_5(OH)_4$] group, which was the main clay mineral found in the WTP sludge [11, 23, 40]. With regards to the calcined sludge (Figure 9b), hematite (Fe_2O_3) and anatase (TiO_2) were recorded in addition to quartz and rutile. Studies [40, 44] using XRD show that wet sludge has a mineralogical composition similar to the clay from the region, with high potential for incorporation into ceramic production.



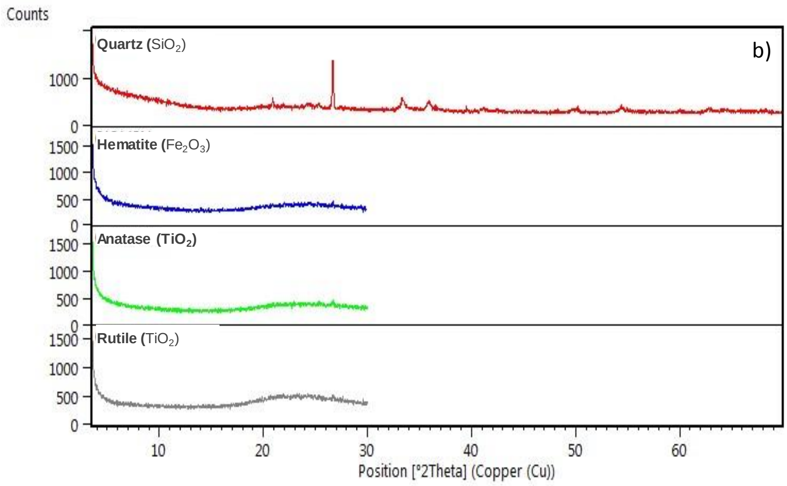


Figure 9: XRD diffractogram a) wet sludge; and b) calcined sludge.

It is important to note that the analysis of mineralogical composition of the sludge by XRD was complex due to the great variation in the mineral components of the WTP sludge, which alternated between crystalline and amorphous. Pinheiro et al. [40] also described difficulties analyzing the sludge by XRD, stating that the majority of the peaks related to the crystalline phases of some minerals (such as ilite and anatase) from the studied sludge, which were confused with the diffractogram background, making interpretation of the results difficult.

In relation to the granulometry characterization of the calcined sludge (Figure 10 and Table 7), 90% of the particles were smaller than 72.44 µm. The equivalent diameter at 50% of accumulated mass was 33.68 µm and the equivalent at 10% was 6.86 µm. The average dimension of the particles was 37.62 µm.

Table 7: Particle diameter of calcined sludge.

AVERAGE DIAMETER	AT 10%	AT 50%	AT 90%	AVERAGE
VALUE (µm)	6.86	33.68	72.44	37.62

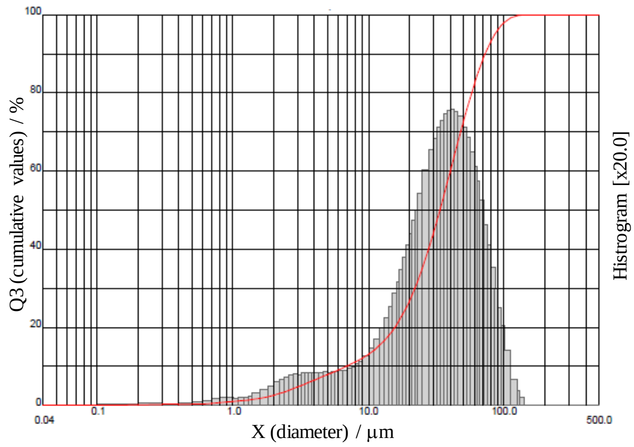


Figure 10: Granulometric distribution of calcined sludge.

Gastaldini et al. [11] found similar particle size distributions in the samples of WTP sludge ash calcined at different temperatures (600ºC and 700ºC) and residence times (1 and 2 hours). For the calcined sludge at 600ºC for 1 hour, the authors observed that 90% of the particles showed sizes smaller than 65.06 µm and the equivalent diameter at 50% of accumulated mass was 20.7 µm [11]. These results were similar to those observed in the present study, however were slightly smaller than the typical dimensions of cement particles. Nonetheless, the authors found satisfactory results from the use of the calcined sludge in replacement of Portland cement in concrete production. The concrete mixes prepared with WTP sludge ash showed increases in strength ranging from 3% to 30% depending on the level replacement and the water/binder ratio used [11].

4. CONCLUSIONS

Characterization of the water sludge produced in a Brazilian WTP was carried out to examine its potential for reuse in concrete production for the civil construction industry.

The wet sludge had a high loss on ignition, which may limit its use. However, the calcination process at 700°C could reduce 90% of material loss on ignition. Calcined WTP sludge had reduced loss on ignition and fine granulometry (average particle diameter of 37.62 µm) due to its chemical characteristics. Under these conditions it may be suitable for use as a raw material in the civil construction industry (cementitious artifacts) since its physicochemical properties were similar to those of other materials used for the same purpose.

The results also indicated that the chemical characteristics of the WTP sludge changed due to seasonal variations. This was highly linked to the influence of climate, rainfall, and soil conditions, as well as the chemical products used in the water treatment process, which highlighted the importance of physical-chemical analysis of the sludge to enable better reuse or disposal.

5. ACKNOWLEDGMENTS

The authors gratefully acknowledge SANEPAR for the opportunity to develop this study and Itaipu Concrete Technology Laboratory (LTCI) for the availability of their laboratories.

6. BIBLIOGRAPHY

[1] CARNEIRO, C., WEBER, P.S., ROSS, B.Z.L., *et al*., “Caracterização do Lodo de ETA gerado no Estado do Paraná”, In: CARNEIRO, C., ANDREOLI, C. V. (Coord.). *Lodo de Estações de Tratamento de Água – Gestão e Perspectivas Tecnológicas*. Curitiba, SANEPAR, pp.131-178, 2013.

[2] SUKSIRIPATTANAPONG, C., HORPIBULSUK, S., BOONGRASAN, S., *et al*., “Unit weight, strength and microstructure of a water treatment sludge–fly ash lightweight cellular geopolymer”, *Construction and Building Materials*, v. 94, pp. 807-816, 2015.

[3] RAMIREZ, K.G., POSSAN, E., DEZEN, B.G., *et al*., “Potential uses of Waste Sludge in Concrete production”, *Management of environmental quality*, v. 28, p. 155-161, 2017.

[4] ANDRADE, C., MYNRINE, V., SILVA, D.A., *et al*., “Compósito para a construção civil a partir de resíduos industriais”, *Matéria*, v. 1, n. 2, pp. 321-329, 2016.

[5] DI BERNARDO, L., DANTAS, A. DB., VOLTAN, P.E.N., *Métodos e Técnicas de Tratamento e Disposição dos Resíduos Gerados em Estações de Tratamento de Água*, São Carlos, LDiBe, 2012.

[6] KONDAGESKI, J.H., CARNEIRO, C., ANDREOLI, C.V., *et al*., “Pesquisas Interdisciplinares e a estruturação dos estudos da Rede Interinstitucional de Pesquisa em Lodo de Água”, In: Carneiro, C.; Andreoli, C. V. (Coord.), *Lodo de Estações de Tratamento de Água – Gestão e Perspectivas Tecnológicas*. Curitiba, SANEPAR, pp. 47-66. 2013.

[7] NÓBREGA, C. C., PEREIRA, S.L.M., BARBOSA, G., *et al*., “Caracterização do lodo residual das lagoas de lodo da estação de tratamento de água – estudo de caso: ETA – Gramame”, In: *Proceedings of 4 Simposio Iberoamericano de Ingeniería de residuos*: *Hacia la sustentabilidad: Los residuos sólidos como fuente de energía y materia prima*, pp. 8-14, México D.F., out., 2011.

[8] SANEPAR. Companhia de Saneamento do Paraná. *Sistema Corporativo de Controle Industrial*. Relatório. Foz do Iguaçu, PR. 2017.

[9] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. NBR 10004:2014 - Resíduos sólidos - Classificação, Rio de Janeiro, 2014.

[10] BRASIL. Lei no 12305, de 02 de agosto de 2010. Institui a Política Nacional de Resíduos Sólidos; altera a Lei no 9.605, de 12 de fevereiro de 1998; e dá outras providências. Diário Oficial da União, Brasília, DF, 13 ago. 2010.

[11] GASTALDINI, A.L.G., HENGEN, M.F., GASTALDINI, M.C.C., et al., “The use of water treatment plant sludge ash as a mineral addition”, *Construction and Building Materials*, v 94, pp. 513–520, 2015.

[12] TSUTIYA, M. T., SERRA, J.C.V., MAFRA, W.A., *et al*. “Aproveitamento e disposição final de lodos de Estações de tratamento de água do estado de São Paulo”, In: 21º Congresso Brasileiro de Engenharia Sanitária e Ambiental, ABES, 2001.

[13] SILVA, É. T., MELO, W.J., TEIXEIRA, S.T., “Chemical attributes of a degraded soil after application of water treatment sludges”, *Scientia Agricola*, v.62, n.6, pp. 559-563, 2005.

[14] BITTENCOURT, S. SERRAT, B.M., AISSE, M.M., *et al*., "Aplicação de lodos de estação de tratamento de água e de tratamento de esgoto em solo degradado", *Engenharia Sanitária Ambiental*, v.17 n.3, pp. 315-324, 2012.

[15] FREITAS, J.G., FILHO, S.S.F. PIVELI, R. P., "Viabilidade técnica e econômica da regeneração de coagulantes a partir de lodos de estações de Tratamento de água", *Revista Engenharia Sanitária Ambiental*, vol.10, n. 2, pp. 137-145, 2005.

[16] CHEN, H., MA, X., DAI, H. "Reuse of water purification sludge as raw material in cement production", *Cement and Concrete Composites*, v. 32, n. 6, pp. 436–439, July 2010.

[17] YEN, C., TSENG, D.H., LIN, T.T., "Characterization of eco-cement paste produced from waste sludges", *Chemosphere*, v. 84, n. 2, pp. 220–226, June 201.

[18] SULLIVAN, C. CHEESEMAN, C.R., GRAHAM, N.J.D., "Disposal of water treatment wastes containing arsenic — A review", *Science of the Total Environment*, v. 408, n. 8, pp. 1770–1778, March 2010.

[19] SOGANCIOGLU, M., YEL, E., SULTAN, U. KESKIN, Y., "Utilization of andesite processing wastewater treatment sludge as admixture in concrete mix", *Construction and Building Materials*, v. 46, pp. 150–155, Set. 2013.

[20] HATVEERA, B., LERTWATTANARUK, P. MAKUL, N., "Effect of sludge water from ready-mixed concrete plant on properties and durability of concrete", *Cement and Concrete Composites*, v. 28, pp. 441-450, 2006.

[21] YAGUE, A., VALLS, S., VÁZQUEZ, E., *et al*., "Durability of concrete with addition of dry sludge from waste water treatment plants", *Cement and Concrete Research*, v. 35, n. 6, pp. 1064–1073, jun. 2005.

[22] TAFAREL, N.F., MACIOSKI, G., CARVALHO, K.Q., *et al*., "Avaliação das propriedades do concreto devido à incorporação de lodo de estação de tratamento de água", *Matéria*, v .21, n. 4, pp. 974-986, 2016.

[23] TARTARI, R., DÍAZ-MORA, N., MÓDENES, A.N., *et al*., "Lodo gerado na estação de tratamento de água Tamanduá Foz do Iguaçu, PR, como aditivo em argilas para cerâmica vermelha: Parte II: incorporação do lodo em mistura de argilas para produção de cerâmica vermelha", *Revista Cerâmica*, v. 57, n. 344, 2011.

[24] MONTEIRO, S.N., ALEXANDRE, J., MARGEM, J.I., *et al*., "Incorporation of sludge waste from water treatment plant into red ceramic", *Construction and Building Materials*, v. 22, n. 6, pp. 1281–1287, jun. 2008.

[25] MARTÍNEZ-GARCIA, C., ELICHE-QUESADA, D., PÉREZ-VILLAREJO, L., *et al*., "Sludge valorization from wastewater treatment plant to its application on the ceramic industry", *Journal of Environmental Management*, vol. 95, Supplement, pp. S343–S348, mar. 2012.

[26] KIZINIEVIC, O., ŽURAUSKIENĖ. R., KIZINIEVIČ, V., *et al*., "Utilization of sludge waste from water treatment for ceramic products", *Construction and Building Materials*, v. 41, pp. 464–473, abr. 2013.

[27] VIEIRA, C. M., MARGEM, J.I., MONTEIRO, S.N., "Alterações microestruturais de cerâmica argilosa incorporada com lodo de ETA", *Matéria*, v.13, n.2, pp.275-281, 2008.

[28] JOHNSON, O.A., NAPIAH, M., KAMARUDDIN, I., "Potential uses of Waste Sludge in Construction Industry: A Review", *Research Journal of Applied Sciences, Engineering and Technology*, v. 8, n. 4, pp. 565-570, 2014.

[29] FADANELLI, L., WIECHETECK , G.K., "Estudo da utilização do lodo de estação de tratamento de água em solo cimento para pavimentação rodoviária", *Revista de Engenharia e Tecnologia*, v. 2, n. 2, Ago, 2010.

[30] UCKER, F. BARROSO, L.B., LOPES, M.I.P., "Utilização do lodo gerado em indústria para a fabricação de argamassa", *Disciplinarum Scientia. Série: Ciências Naturais e Tecnológicas*, vol. 11, n. 1, pp. 106-114, 2010.

[31] RODRIGUEZ, N.H., MARTÍNEZ RAMÍREZ, S., BLANCO VARELA, M.T., *et al*., "Re-use of drinking water treatment plant (DWTP) sludge: Characterization and technological behaviour of cement mortars with atomized sludge additions", *Cement and Concrete Research*, v. 40, n, 5, pp. 778–786, maio 2010.

[32] HOPPEN, C., PORTELLA, K.F., JOUKOSKI, A., *et al*., "Co-disposição de lodo centrifugado de Estação de Tratamento de Água (ETA) em matriz de concreto: método alternativo de preservação ambiental", *Revista Cerâmica*, v. 51, n. 318, pp. 85-95, Jun. 2005.

[33] WANG, Q., WEI, W., GONG,Y., *et al*., "Technologies for reducing sludge production in wastewater treatment plants: State of the art", *Science of The Total Environment*, v. 587, pp. 510-521, 2017.

[34] GOOGLE EARTH. Foto de satélite de Foz do Iguaçu, Paraná Brasil. Disponível em: http://earth.google.com. Acesso: 12 de janeiro de 2014.

[35] RAMIREZ, K.G., *Viabilidade do aproveitamento de Resíduo de Estação de Tratamento de Água (ETA) na Confecção de Concretos,* Dissertação de M.Sc., PPGETamb/UTFPR, Medianeira, PR, 2015.

[36] APHA, AWWA, and WEF. Standard Methods for the Examination of Water and Wastewater, 21st Ed. American Public Health Association, Washington, D. C. 2005.

[37] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. NBR 10005: Procedimento para obtenção de extrato lixiviado de resíduos sólidos, 2004.

[38] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. NBR 10664: Águas – Determinação de resíduos (sólidos) – método gravimétrico. Rio de Janeiro: 1989.

[39] RICHTER, C.A. *Tratamento de Lodos de Estações de Tratamento de Água*. São Paulo: Editora Edgard Blücher LTDA, 2001.

[40] PINHEIRO, B. C. A., ESTEVÃO, G. M., SOUZA, D. P. “Lodo proveniente da estação de tratamento de água do município de Leopoldina, MG, para aproveitamento na indústria de cerâmica vermelha Parte I: caracterização do lodo”, *Matéria*, v. 19, n. 3, pp. 204-211, 2014.

[41] LIN, C., WU C.H., HO, H.M., “Recovery of municipal waste incineration bottom ash and water treatment sludge to water permeable pavement materials”, *Waste Management*, v. 26, n. 9, pp. 970–978, 2006.

[42] WOLFF, E., SCHWABE, W. K., CONCEIÇÃO, S. V. “Utilization of water treatment plant sludge in structural ceramics”, *Journal of Cleaner Production, v* 96, pp. 282e289, 2015.

[43] ASSOCIAÇÃO BRASILEIRA DE NORMAS TÉCNICAS. NBR 12653: Materiais pozolânicos - Requisitos. Rio de Janeiro, 2012.

[44] TEIXEIRA, S.R. SANTOS, G.T.A., SOUZA, A.E., *et al*., “The effect of incorporation of a Brazilian water treatment plant sludge on the properties of ceramic materials”, *Applied Clay Science*, v. 53, n. 4, pp. 561–565, out. 2011.